भारतीय मानक
Indian Standard

IS 1448 [P : 43] : 2018

पेट्रोलियम तथा उसके उत्पादों के परीक्षण की पद्धति

[पी : 43]

रंग सूचक पद्धति द्वारा ब्रोमीन संख्या

(तीसरा पुनरीक्षण)

Methods of Test for Petroleum and its Products

[P : 43]

Bromine Number by Colour Indicator Method

(Third Revision)

ICS 75.160.20

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Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard [P : 43] (Third Revision) was adopted by the Bureau of Indian Standards on recommendation of the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was earlier revised in 1991. In this revision, Amendment no 1 (issued in 2011) to the second revision was included and references are updated. The solvent, 1,1,1–trichloroethane was replaced with dichlormethane, as 1,1,1-trichloroethane is an ozone depleting substance. Comparative work had shown that its replacement by Dichloromethane gives no discernible change in results and thus the replacement of solvent has been made.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 43]

BROMINE NUMBER BY COLOUR INDICATOR METHOD

(Third Revision)

1 SCOPE

1.1 This standard [P : 43] covers the test method for the determination of the bromine number of liquid petroleum products substantially free from butanes and butenes. It is generally applicable to liquid petroleum products that fall in the following limits:

90 Percent Distillation Point, °C	*Bromine Number Max (see Note)*
Under 205	115
205 to 327	15

NOTE — These limits are imposed since true precision of the method has been determined only up to these bromine numbers.

1.1.1 The bromine number does not correspond necessarily to a true content of un saturates, since sulphur compounds and certain polycyclic aromatic hydrocarbons are reported to react with bromine.

1.2 The reported behavior of the main hydrocarbon types is as follows:

Hydrocarbon Type	*Level of Bromine obtained with Pure Reference Compounds*
Paraffins	Zero bromine number
Straight-chain Olefins	Theoretical
Branched-Chain olefins	Mostly higher than theoretical
Cyclic olefins	Theoretical
Non-conjugated Dienes	Not assessed
Non Conjugated Cyclic dienes	Theoretical
Conjugated Dienes	Lower than theoretical
Aromatics with Unsaturated side chains	Not Assessed
Aromatics Monocyclic	Zero Bromine Number
Aromatics Bicyclic	Zero Bromine Number
Aromatics, Polycyclic	Mostly Higher than theoretical
Naphthenes	Zero Bromine Number

2 REFERENCE

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
695 : 1986	Specification for acetic acid (*third revision*)
1448 [P : 44] : 2013/ ISO 3839 : 1996	Methods of test for petroleum and its products: Part 44 Determination of bromine number of distillates and aliphatic olefine — Electrometric method

3 TERMINOLOGY

For the purpose of this method, the following definition shall apply.

3.1 Bromine Number — The number of grams of bromine consumed by 100 g of the sample when reacted under conditions specified in this method.

4 OUTLINE OF THE METHOD

The sample, dissolved in dichloromethane, is treated at room temperature with an excess of bromide-bromate solution in the presence of glacial acetic acid. The excess bromine is reduced with potassium iodide and the liberated iodine determined by titration with sodium thiosulphate solution.

5 APPARATUS

5.1 Iodine Number Flasks — 500 ml, glass stoppered.

5.2 Volumetric Flasks — One mark flasks of 50 ml and 1 000 ml capacity class A.

5.3 Pipettes — 5 ml capacity, Class A. Graduated Pipettes of between 2 ml and 25 ml as required by the test portion volumes.

6 REAGENTS

6.1 Dichloromethane (CH_2Cl_2) — Solvent in which sample is dissolved at room temperature.

6.2 Acetic Acid, Glacial — 99 percent, m/m

6.3 Sodium Thiosulphate Solution — Approximately 0.1 N accurately standardized. Dissolve 25 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_20$) in water and add 0.01 g of sodium carbonate (Na_2CO_3) to stabilize the solution. Dilute to 1 litre and mix thoroughly by shaking. Standardize by any accepted procedure that determines the normality with an error ± 0.0002. Restandardize at intervals frequent enough to detect changes of ± 0.0005 in normality.

6.4 Potassium Iodide Solution — 150 g per litre.

6.5 Starch Solution — 0.5 percent. Triturate 1 g of soluble starch with 5 to 6 ml of boiling water. Add the suspension to 1 litre of distilled water and boil for 3 minutes. Allow to cool and decant the clear supernatant liquid into glass stoppered bottle. Prepare fresh daily.

6.6 Potassium Bromide — Bromate Solution — Standard 0.5 N. Weigh out exactly 51 g of potassium bromide and 13.92 g of potassium bromate, dissolve in distilled water and dilute the solution to 1 litre.

7 PROCEDURE

7.1 Place 10 ml of dichloromethane (CH_2Cl_2) in a 50 ml volumetric flask and by means of a pipette, introduce a quantity of sample as indicated in Table 1 (*see* Note 1). Either obtain the mass of sample introduced by difference between the mass (to the nearest mg) of the flask before and after addition of sample or if the density is known accurately, calculate the mass from the measured volume. Fill the flask to the mark with dichloromethane (CH_2Cl_2) and mix well (*see* Note 2). Pipette a 5 ml aliquot of the solution into a 500 ml iodine number flask containing 50 ml of glacial acetic acid.

NOTES:

1 Frequently, the order of magnitude of the bromine number of sample is unknown. In this case, a trial test is recommended using a 2 g of sample in order to obtain the approximate magnitude of the bromine number. This explanatory test is to be followed with another determination using, the appropriate sample size as indicated in Table 1.

2 The dilution of the sample may be made with glacial acetic acid in place of dichloromethane (CH_2Cl_2). If desired .If the dilution of the sample is made with glacial acetic acid, the 5 ml aliquot should be added to an iodine number flask containing 45 ml of glacial acetic acid and 5 ml of dichloromethane (CH_2Cl_2).

Table 1 Sample Size

Bromine Number	Sample Size, g
0-10	20-16
Over 10-20	10-8
Over 20-50	5-4
Over 50-100	2-1.5
Over 100	1-0.5

7.2 Shield the flask from exposure to direct sunlight and keep it at a temperature of 20 ± 5 °C. Add bromide – bromate solution from a burette at a rate of 1-2 drops per second swirling the contents of the flask constantly during the addition. Continue adding the reagent until the reaction mixture assumes a yellow color (*see* Note 1) that remains for at least 5 s. Add an additional 1 ml of the reagent as quickly as possible, stopper the flask and continue swirling immediately for 40 ± 5 s. At the end of this period place 5 ml of potassium iodide solution in the lip of the flask and lift the stopper, allowing the solution to flow slowly into the flask. Replace the stopper, shake vigorously, add 100 ml of water and shake again for one minute. Titrate promptly with thiosulphate solution. Near the end of the titration, add 1 ml of starch indicator solution and titrate slowly to disappearance of the blue color. The final drop shall be that which removes the blue color for not less than 30 s. Discard the test if the back titration is less than 5 ml or greater than 10 ml of thiosulphate solution (*see* Note 2).

NOTES:

1 The yellow colour produced should be equivalent to that obtained by adding 0.5 ml bromide solution to 50 ml glacial acetic acid and 5 ml of dichloromethane (CH_2Cl_2) in a similar flask.

2 In certain cases, notable high boiling samples, it is impossible to meet the requirements of 5 ml to 10 ml thiosulphate back titration, in such cases, the result are indeterminate.

Make duplicate blank determinations on each lot of reagents by repeating the entire procedure but using 5 ml of dichloromethane (CH_2Cl_2) in place of the sample (*see* Note).

NOTE — In routine work no correction need be applied so long as the blank does not exceed 0.02 milli- equivalent of bromine. For work of highest accuracy, correction should be applied if the blank exceeds 0.01 milli – equivalent. The correction shall be made by substracting from the term (VM – vm) in the dichloromethane (CH_2Cl_2) actually present in the 5 ml aliquot of sample solution used in the analysis. This volume may vary from 2.5 to 4.9 ml, depending upon the quantity of sample used.

8 CALCULATION AND REPORTING

Calculate the bromine number of the sample as follows:

Bromine number = 7.99 (VN – vn)/M

Where,

V = volume in ml, of bromide-bromate solution added to the sample;

N = normality of the bromide-bromate solution;

v = volume in ml, of sodium thiosulphate solution used for the back titration;

n = normality of the sodium thiosulphate solution; and

M = mass in g, of sample represented by the aliquot used.

Report the results to the nearest 0.1 as bromine number.

NOTE — The approximate relationship between the bromine number [P : 43] of straight run gasoline, kerosenes and light gas oils determined by this standard and those determined by the electrometric method is given by the equation.

(Results of P : 43)$^{1/3}$ = 1.012 (Results of P : 44 (Bromine number by electrometric method)$^{1/3}$ + 0.135.

9 PRECISION

Result of duplicate tests shall not differ by more than the following amounts:

90 Percent Distillation Point, °C	Range of Bromine Number	Repeatability	Reproducibility
Under 205	0 to 115	$0.15M^{2/3}$	$0.31M^{2/3}$
206-327	0 to 15	$0.18M^{2/3}$	0.51M*

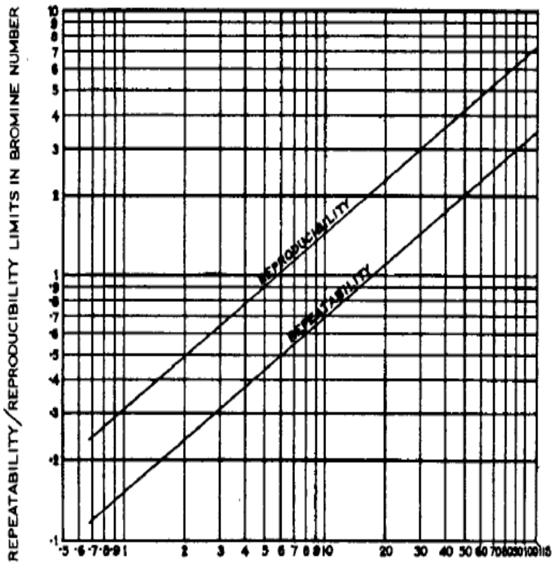

Fig. 1 Precision values for High Volatility Samples

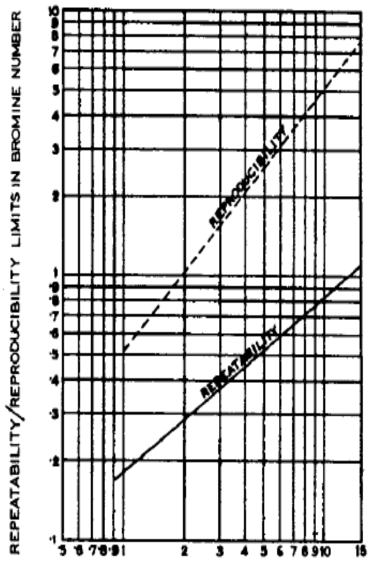

Fig. 2 Precision values for Lower Volatility Samples

where, M is the mean of duplicate results.

* Provisional results obtained from a limited amount of data.

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Amendments Issued Since Publication

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